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METHOD FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY MIXTURES OF REGENERATED CELLULOSE FIBRES AND COTTON

PART II CADOXEN SOLVENT METHOD

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METHOD FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY MIXTURES OF REGENERATED CELLULOSE FIBRES AND COTTON

PART II CADOXEN SOLVENT METHOD

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METHOD FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY MIXTURES OF REGENERATED CELLULOSE FIBRES AND COTTON

PART II CADOXEN SOLVENT METHOD

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 12 March 1976, after the draft finalized by the Chemical Methods of Test Sectional Committee, had been approved by the Textile Division Council.

0.2 The Indian Standard on method for quantitative chemical analysis of binary mixtures of regenerated cellulose fibres and cotton, when first published in 1962 covered only sodium zincate method. While taking up the revision of this standard, the Committee decided to cover Cadoxen solvent method and formic acid-zinc chloride method also as Part II and Part III of this standard respectively.

0.3 The use of different fibre blends in textiles has necessitated the formulation of standard methods for identification and quantitative estimation of respective fibres. The quantitative analysis of textile fibres in mixtures is of considerable importance to textile technologists, traders and consumers.

0.4 In the preparation of this standard (Parts I to III) due weightage has been given to the test methods prevalent in the industry and trade and also the methods developed by International Organization for Standardization, ISO. Parts I and III are based mainly on ISO 1833-1973 'Textiles — Binary fibre mixtures — Quantitative chemical analysis'. This part (Part II) is based on the method developed by Prof W. B. Achwal of the University of Bombay (Department of Chemical Technology), Bombay, and the extensive experiments carried out at the Ahmedabad Textile Industry's Research Association, Ahmadabad, the Bombay Textile Research Association, Bombay, and the Silk & Art Silk Mills' Research Association, Bombay.

0.5 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part II) prescribes Cadoxen solvent method for quantitative chemical analysis of binary mixtures of regenerated cellulose fibres and cotton in any textile form, such as fibre, yarn or fabric.

NOTE — Before conducting an analysis according to this method, the fibres present in the mixture should be identified (*see* IS : 667-1955†) and the sample to be analysed shall be freed from all non-fibrous matter by the method given in Appendix A. Dye in the dyed fibres is considered to be an integral part of the fibre and is not to be removed.

1.1.1 This method is not applicable to mixtures in which the cotton has suffered extensive chemical degradation, nor when the viscose, cupro or modal fibre is rendered incompletely soluble by the presence of certain permanent finishes or reactive dyes that cannot be removed completely.

2. PRINCIPLE

2.1 A sample of the mixture is dried and weighed. The regenerated cellulose fibres in the sample are dissolved in Cadoxen solvent. The residue, that is, cotton, is collected, washed, dried and weighed and the proportion of regenerated cellulose and cotton is calculated.

3. SAMPLING

3.1 Lot — The quantity of textile material of definite type and quality delivered to one buyer against one despatch note shall constitute a lot.

3.1.1 If the textile material is fibre or yarn and the lot consists of more than 200 kg of the material, it shall be divided into sub-lots weighing 200 kg or less and each sub-lot shall be tested separately.

3.2 Sampling for Fibre and Yarn — From a sub-lot 15 increments weighing approximately 10 g each shall be taken from different parts and mixed thoroughly to constitute a **test sample.**

3.3 Sampling for Fabrics — From each piece in the gross sample selected as in IS : 5463-1969‡ cut out small portions from at least two different parts weighing about 25 g. Dissect the small portions of the fabric thus collected into yarn and mix thoroughly. This shall constitute a **test sample.**

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*Rules for rounding off numerical values (*revised*).

†Simple methods for identification of common commercial textile fibres.

‡Methods for sampling of cotton fabrics for chemical tests.

4. APPARATUS

4.1 Sintered Glass Filter Crucibles — of appropriate capacity with pore size of 90 to 150 microns (porosity 1) and fitted with ground glass stoppers. If the stoppers are not available, the crucibles should be enclosed in weighing bottles for weighing.

4.2 Desiccator — with phosphorus pentoxide as desiccant.

4.3 Analytical Balance — of an accuracy of 0·000 2 g.

4.4 Conical Flask — of 150 ml capacity, provided with a glass stopper.

4.5 Ventilated Oven — for drying specimens/residue at 105 ± 3°C.

5. REAGENTS

5.1 Cadoxen Solvent — containing 4·6 ± 0·1 percent cadmium and 28 percent ethylenediamine.

5.1.1 *Method of Preparation* — Dilute freshly distilled ethylenediamine with distilled water to 28 percent (*m/m*). Cool the solution below 0°C. To this slowly add 8 percent cadmium oxide under vigorous stirring over a period of 4 hours, after which continue stirring for 2 hours. Keep the solution in cold storage (0 to 5°C) for a period of 60 hours and then slowly bring it to room temperature (30°C). Filter the solvent through sintered glass crucible and analyse for ethylenediamine and cadmium contents as given in Appendix B.

5.2 Light Petroleum — boiling range 40 to 60°C.

6. PREPARATION OF TEST SPECIMENS

6.1 From each sample (**3.2** or **3.3**), after removing size and finishes as prescribed in Appendix A, draw a representative specimen weighing about 2 to 3 g. Cut the test specimen into pieces of approximately 25 mm length.

7. PROCEDURE

7.1 Take about 0·25 g of the specimen in weighing bottle and dry at 105 ± 3°C for 3 hours, cool over phosphorus pentoxide in desiccator and weigh. Transfer the contents to 150-ml stoppered flask and determine the exact mass of the specimen by weighing the bottle again.

7.2 Add 50 ml of Cadoxen solvent to the flask and shake the flask at room temperature for 1 hour. Filter the contents through a weighed sintered glass crucible.

7.3 Wash the residue (undissolved cotton) with 10 ml of Cadoxen solvent, and then give several washes with distilled water till the washings are free of cadmium. Dry the crucible with residue at 105 ± 3°C for 3 hours, cool over phosphorus pentoxide in desiccator and weigh. Determine the mass of the residue.

7.4 Similarly carry out the test for other test specimens.

8. CALCULATION

8.1 Calculate the percentage of cotton fibre in the specimen by the following formula for each test specimen and find out the average of all the values:

$$\text{Percentage of cotton} = \frac{100 \times m_r}{m_s}$$

where

m_r = mass in grams of the residue (**7.3**), and

m_s = mass in grams of the specimen (**7.1**).

NOTE — Since cotton fibre does not undergo any loss in mass under above conditions, no correction factor is necessary.

9. REPORT

9.1 The report shall include the following information:

a) Type of the material,
b) Percentages of regenerated cellulose and cotton in the mixture, and
c) Number of specimens tested.

APPENDIX A

(*Note under Clause* 1.1 and *Clause* 6.1)

METHOD FOR REMOVAL OF SIZE AND FINISHES FROM TEST SAMPLES

A-1. From each test sample, draw a representative sample weighing about 10 g and dry it at 105 ± 3°C for 3 hours. Treat the sample as given in **A-2** to **A-6**. However, if the nature of the finish present in the sample is known only relevant steps may be carried out.

A-2. Extract the sample with carbon tetrachloride in a soxhlet apparatus for 2 hours at a minimum rate of 6 cycles per hour. (This removes oils, fats, waxes, certain thermoplastic resins, etc.)

A-3. Extract the sample with ethyl alcohol in a soxhlet apparatus for 2 hours at a minimum rate of 6 cycles per hour. (This removes soaps, cationic finishes, etc.)

A-4. Treat the sample with 200 ml of water at 50°C for 30 minutes, stirring occasionally with glass rod or mechanically. Rinse thrice with fresh portion of warm water (50°C) and dry. (This removes water-soluble materials.)

A-5. Give an enzyme treatment to the sample under conditions depending upon the nature of enzyme used and rinse thoroughly. (This removes starch, gelatine, etc.)

A-6. Immerse the sample in 200 ml of 0·1 N hydrochloric acid at 18°C for 25 minutes, stirring gently every 3 minutes. Rinse thoroughly with water at 80°C containing a few drops of ammonia and then finally with plain water. Remove excess water from the sample by squeezing, suction or centrifuging and allow to dry. (This removes resins.)

APPENDIX B

(*Clause* 5.1.1)

METHODS FOR DETERMINATION OF ETHYLENEDIAMINE AND CADMIUM CONTENTS IN CADOXEN SOLVENT

B-1. DETERMINATION OF ETHYLENEDIAMINE CONTENT

B-1.1 Weigh accurately about 5 g of the Cadoxen solvent, dilute in water and make up to 100 ml in a volumetric flask (*solution A*).

B-1.2 Titrate a 20-ml aliquot of *solution A* prepared as in **B-1.1** with 0·5 N sulphuric acid in the presence of methyl orange indicator.

B-1.3 Calculation

$$\text{Ethylenediamine, percent by mass} = v \times N \times \frac{100}{20} \times \frac{100}{m} \times \frac{30}{1\,000}$$

$$= \frac{v \times N \times 15}{m}$$

where

v = volume in millilitres of sulphuric acid consumed,
N = normality of sulphuric acid, and
m = mass in grams of the sample taken for the test.

B-2. DETERMINATION OF CADMIUM CONTENT

B-2.1 Pipette out 20 ml of *solution A* (**B-1.1**) and add two-thirds the quantity of 0·5 N sulphuric acid required in titration as in **B-1.2** in order to keep *p*H at a proper level for getting a sharp end-point. Add a pinch of indicator mixture (2·5 g eriochrome black T + 1·0 g methyl red + 200·0 g sodium chloride) and 2 ml of buffer solution (350 ml ammonia, 25 percent + 54 g ammonium chloride). Add 20 ml of distilled water and titrate the mixture with 0·05 M EDTA solution to be standardized against 0·05 M cadmium acetate (analytical reagent grade) till the colour changes from wine-red to bright green.

B-2.2 Calculation

$$\text{Cadmium content, percent by mass} = v \times M \times \frac{100}{20} \times \frac{100}{m} \times 0{\cdot}112\,4$$

$$= v \times M \times \frac{56{\cdot}2}{m}$$

where

v = volume in millilitres of 0·05 M EDTA solution consumed,
M = molarity of EDTA solution, and
m = mass in grams of the sample taken for the test.

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